INFLUENCE OF TEMPERATURE AND CONCENTRATION OF REACTING SOLUTION ON MERCERISATION OF RAW JUTE FIBRE*

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(Plates IVA and IV B)

ABSTRACT. Raw jute fibres were treated with caustic soda solutions of various concentrations (5% to 50%) at different temperatures (−10°C to +60°C), washed with water and dried, and the products thus obtained were analysed by studying X-ray diffraction photographs which revealed three distinct types of patterns, *e.g.*, (*i*) some due to native cellulose (*ii*) some due to partially mercerised cellulose and (*iii*) the rest due to completely mercerised cellulose. The results obtained have been given graphically. It is pointed out that the results obtained with raw jute fibre differ considerably from those obtained in the case of cotton by Sisson and Saner, especially in the higher concentration-region, *e.g.*, concentration between 40% and 50% and temperature range −10°C to +40°C. An attempt has been made to explain these differences in the two cases.

INTRODUCTION

It is well known that when caustic soda solution reacts with pure cellulose, soda cellulose is formed and on washing the product with water and drying it a final product is obtained which has the same chemical constitution as that of cellulose but has a crystal structure quite different from that of native cellulose. This product is called hydrated cellulose. The percentage of cellulose thus changed depends however on the concentration and temperature of the reacting alkali solution. This problem was first investigated in the case of cotton cellulose by Sisson and Saner (1941). It was shown recently by Sirkar and Saha (1947) that the crystal structure of the hydrated cellulose obtained similarly from raw jute fibre is different from that of the hydrated cellulose obtained from cotton. Also for a particular concentration of the solution at a particular temperature the percentage of hydrated cellulose formed in this way in the case of jute fibre seemed to be different from that observed in the case of cotton fibre by Sisson and Saner. It was therefore thought worth-while to investigate the problem more thoroughly with raw jute fibre as the starting material and by using NaOH solutions of different strengths at different temperatures to see if there is any influence of the high percentage of lignin present in raw jute fibre on the mechanism of mercerisation. The X-ray analysis of the hydrated cellulose can give the information

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whether the sample consists wholly of hydrated cellulose or partly of some native cellulose, because the positions of 101 and 10$\bar{1}$ reflections in the former case is quite different from those in the latter. Hence this method has been employed in the present investigation and the results which have been discussed later are found to be different from those observed in the case of cotton cellulose by Sisson and Saner.

EXPERIMENTAL

Raw jute fibres cut to small lengths (about 7 cm.) were first cleaned and dipped into about 10 c.c. of caustic soda solution of known strength in a test tube previously brought to the desired temperature by keeping the test tube partly immersed in a water bath hot or cold, or in a freezing mixture as the case might be. No tension was applied during the treatment. After treatment at a definite temperature for about 10 minutes the sample was taken out and washed for about an hour with water at the same temperature. When the temperature of NaOH solution used was below 0°C the washing was effected with ice-cold water. The sample of fibre was then dried in air for about two days. X-ray photographs of these samples were taken after making all the strands parallel by pressing them mildly and holding taut during the exposure in a specially designed camera used previously by Sirkar and Saha (1947). The photographs were taken with a very fine slit of 0.5 mm. bore and 5 cm. in length using CuKα radiation from a Hadding tube. The patterns were examined visually and by noting the absence or presence of 101 and 10$\bar{1}$ reflections of the native cellulose, it was ascertained whether native cellulose was absent or partially present in the sample. The results observed in this way are given in Table I and they are represented graphically in Fig. 1. Some of the diffraction patterns are reproduced in Plates IVA and IVB.

RESULTS AND DISCUSSION

The hatched portion in Fig. 1 corresponds to reactions which produce cent per cent hydrated cellulose, the dotted portions represent the conditions under which the product contains some percentage of native cellulose besides hydrated cellulose, the clear portions represent the conditions under which the reacting solution had no effect on the native cellulose. Owing to experimental difficulties it was not possible to use solutions at temperatures lower than $-10°C$. For comparison the curve obtained with raw cotton fibre by Sisson and Saner (1941) is reproduced in Fig. 2.

It can be seen from Table I that the degree of mercerisation depends in a peculiar way on the temperature and concentration of the solution used. Further, the results of the present investigation, given in Table I and graphically represented in Fig. 1, differ appreciably from those obtained in case of cotton by Sisson and Saner, as can be seen from a comparison of the curves

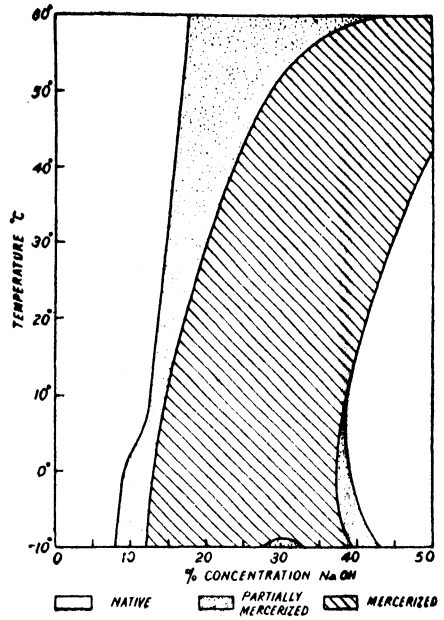
60°
50°
40°
30°
20°
10°
0°
-10°
TEMPERATURE °C
0
10
20
30
40
50
% CONCENTRATION NaOH
NATIVE
PARTIALLY MERCERIZED
MERCERIZED

FIG. 1

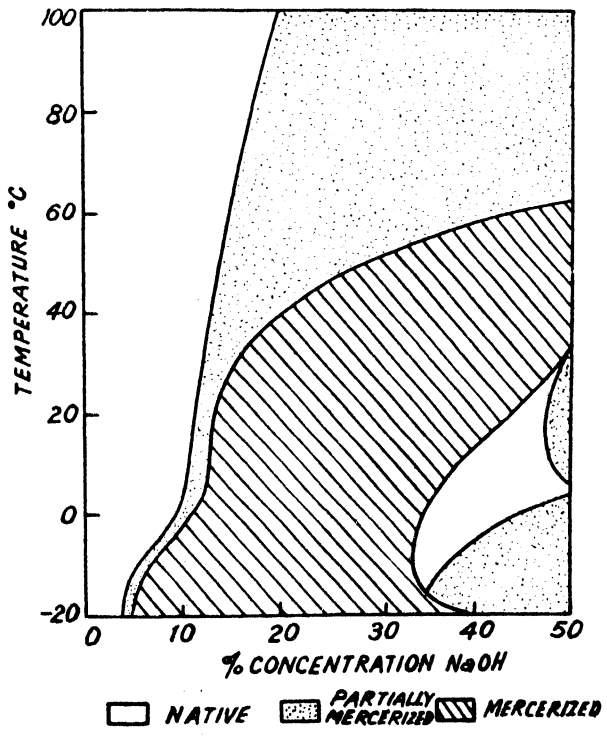
100
80
60
40
20
0
-20
TEMPERATURE °C
0
10
20
30
40
50
% CONCENTRATION NaOH
NATIVE
PARTIALLY MERCERIZED
MERCERIZED

FIG. 2

TABLE I

Strength of NaOH Soln.	Temp. of the solution in °C	Degree of mercerisation	
		Present author	Sisson and Saner
50%	60°	Complete	Complete
50%	40°	Native	Complete
50%	20°	Native	Partial
50%	0°	Native	Partial
50%	−10°	Native	Partial
45%	60°	Complete	Complete
45%	40°	Complete	Complete
45%	20°	Native	Native
45%	0°	Native	Native
45%	−10°	Native	Partial
40%	60°	Partial	Partial
40%	40°	Complete	Complete
40%	20°	Complete	Complete
40%	10°	Native	Native
40%	0°	Native	Native
40%	−10°	Partial	Partial
35%	60°	Partial	Partial
35%	40°	Complete	Complete
35%	15°	Complete	Complete
35%	−10°	Complete	Native
30%	60°	Partial	Partial
30%	40°	Complete	Complete
30%	15°	Complete	Complete
30%	−10°	Partial	Complete
20%	50°	Partial	Partial
20%	25°	Complete	Complete
20%	15°	Complete	Complete
20%	−10°	Complete	Complete
10%	5°	Native	Partial
10%	0°	Partial	Partial
10%	−10°	Partial	Complete
5%	0°	Native	Native
5%	−10°	Native	Partial

in Figs. 1 and 2. For instance, Fig. 1, shows that the range of complete mercerisation at a low temperature of about −10°C extends from 12% to 38% with a small hump of partial mercerisation at 30% NaOH solution, whereas in cotton cellulose (Fig. 2) it was found to extend from 7% to 34%. The range of partial mercerisation (Fig. 1) which is preceded by the complete mercerisation zone and followed by a native zone begins from 8% to 9% at −10°C whereas in case of cotton it begins from 5% at the same temperature. Sisson and Saner obtained the mercerisation of cotton at 20°C when the concentration of the alkali used was about 13% to 14%, but in the case of jute fibre only partial mercerisation is observed with this strength and for complete mercerisation at 20°C the strength of the NaOH solution required is about 18%. It is evident from Fig. 1 that in the reactions at low temperature the concentration of the alkali solution required for complete mercerisation is higher than that in the case of cotton and below a strength of 12% of the solution it is not possible to obtain completely mercerised sample of jute fibre even when the temperature of the reacting solution is −10°C, but in the case of cotton even a 7% solution gives complete mercerisation at this temperature. It is thus seen that in the temperature range from −10°C to +40°C, the concentration required for obtaining totally mercerised sample from raw jute fibre at a particular temperature is higher than that required in the case of cotton. From 50°C up to about 60°C, however, both cotton fibre and raw jute fibre yield the same results.

It also appears from Fig. 1 that at higher concentration, *e g.*, about 48% the complete mercerisation of raw jute fibre cannot be effected if the temperature of the solution used is below 40°C In the case of cotton complete mercerisation with the same solution is obtained above 30°C. But while in this latter case the zone of partial mercerisation is larger than that of native cellulose reverse is true in the case of raw jute fibre at this high concentration range. It is found that with 40% and 45% NaOH solutions the temperature of the solution should be raised above 15°C and 30°C respectively for obtaining complete mercerisation in the case of raw jute fibre.

Again, the examination of Fig. 2 shows that in the case of cotton the mercerisation is complete with 20% solution in the temperature range between −20°C and +40°C, with 30% solution between −20°C and +52°C and with 40% solution the mercerisation does not begin below 16°C and from 16°C to about 58°C the mercerisation is complete, whereas in case of jute the corresponding temperature ranges for complete mercerisation are −10°C to +30°C, −8°C to +52°C, and +15°C to +59°C respectively.

From the results observed it is seen that the process of mercerisation is facilitated when the condition of the treatment is such that low temperatures are maintained when the concentrations of the NaOH solution used are low and high temperatures when the concentrations are high, though the complete mercerisation zone at high temperature and high concentration is small. It appears from above that concentration of alkali is not the only factor influ-

encing the process of mercerisation. It has been suggested by Sisson and Saner (1941) that in the case of cotton the failure of complete mercerisation in the reactions of high concentrations (above 40%) and low temperatures is probably due to the formation of sodium hydroxide hydrates which are unable to penetrate into the micelles. If this be true it would be more difficult for these hydrates to enter into the micelles when the latter contain cements of lignin. Thus the want of any change in the cellulose in jute fibre in reactions with concentrated NaOH solutions at temperature below 40°C, observed in the present investigation seems to support the hypothesis put forward by Sisson and Saner. But this hypothesis cannot account for all the results obtained. An alternative hypothesis, therefore, is being put forward below.

Mercerisation being an exothermic process, the formation of native cellulose with NaOH solutions of lower concentration at high temperature can be easily understood, because exothermic processes are not favoured by high temperatures according to Le Chatelier and Braun's Principle. But the absence of mercerisation with alkali solution of high concentration at lower temperature is not explicable by the above principle. It is probable that at higher concentrations of alkali an impervious surface-coating of the product is formed which prevents the diffusion (Saha, 1947) of the alkali inside the micelles. This coating may be dissolved when dilute solutions are used but it seems that it is dissolved only at higher temperature when a concentrated solution is used. The low range of mercerisation at this high concentration of alkali solution when the temperature is also high, is obviously due to the exothermic nature of reaction.

On comparison of Fig. 1 with Fig. 2 it is observed that both the partial and complete mercerisation zones at low concentration and low temperatures have shifted towards the higher concentration side. It is also observed that between concentration range of 45% and 50% the partial mercerisation zone present in cotton cellulose is totally absent in case of raw jute fibres. It appears that the differences in these two cases may be due to the presence of lignin in the raw jute fibre. In dilute solutions of NaOH, some portion of NaOH solution is spent up in reacting with lignin and therefore only a small fraction of cellulose is reacted upon. At high concentration it appears that lignin facilitates the formation of a more impervious coating on its surface and thus prevents the alkali solution to react with the cellulose. It seems that this coating is soluble at temperatures above 40°C and therefore complete mercerisation of the fibre is effected at higher temperatures.

ACKNOWLEDGMENT

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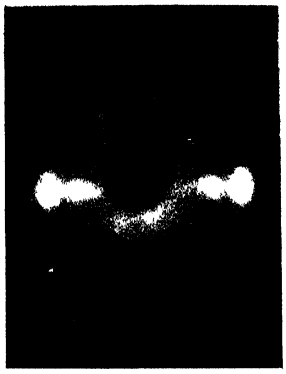

Fig 3

Fig. 4

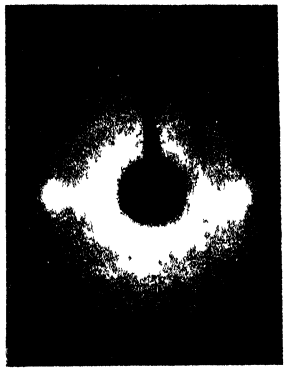

Fig. 5

Fig. 6

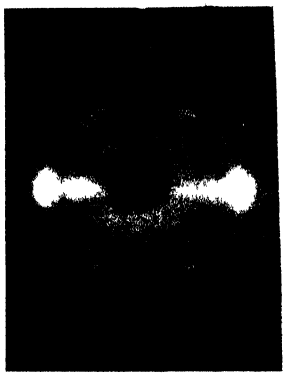

Fig 7

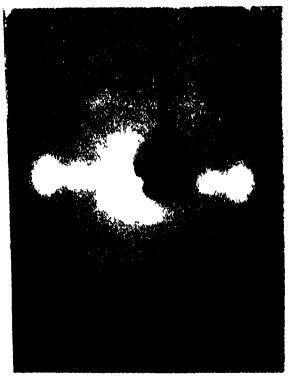

Fig. 8

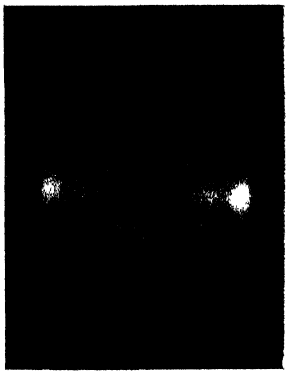

Fig. 9

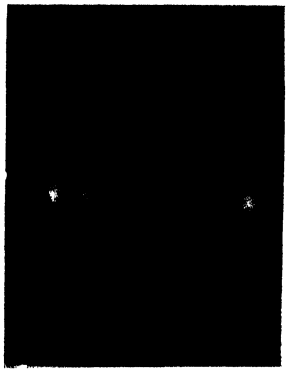

Fig. 10

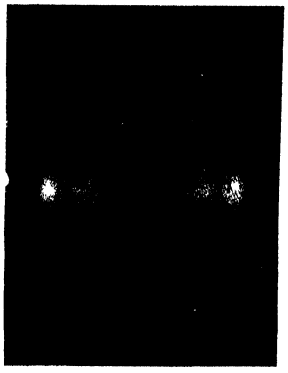

Fig 11

X-ray diffraction patterns.

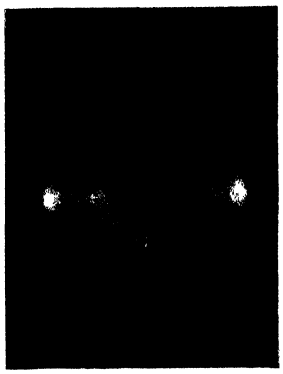

Fig. 12

Fig. 13

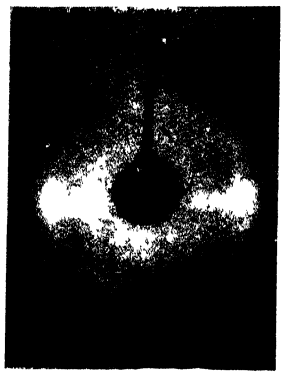

Fig. 14

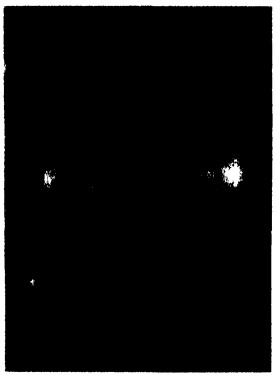

Fig. 15

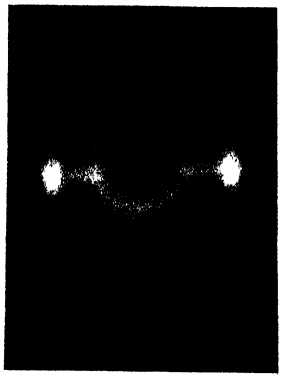

Fig. 16

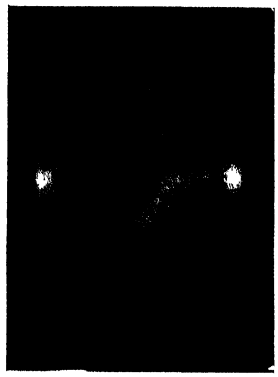

Fig. 17

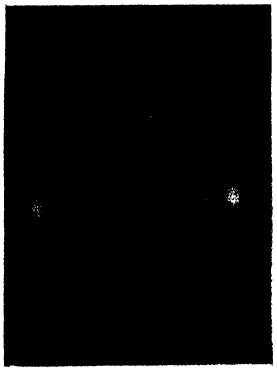

Fig 18

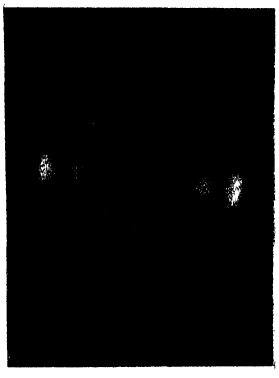

Fig. 19

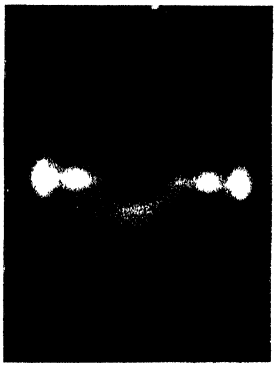

Fig. 20

X-ray diffraction patterns.

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EXPLANATION OF PLATES

Plate IV-A

Fig. 3. Raw jute fibre.
Fig. 4. Treatment with NaOH solution of 50% at 60°C
Fig. 5. ,, , , 50% at 40°C.
Fig. 6. ,, ,, ,, 50% at 20°C.
Fig. 7. ,, ,, ,, 50% at −10°C.
Fig. 8. ,, ,, ,, 15% at 0°C.
Fig. 9. ,, ,, ,, 40% at 60°C.
Fig. 10. ,, ,, ,, 40% at 10°C.
Fig. 11. ,, ,, ,, 40% at −10°C.

Plate IV-B

Fig. 12. Treatment with NaOH solution of 35% at −10°C.
Fig. 13. ,, ,, ,, 35% at 60°C.
Fig. 14. ,, ,, ,, 30% at 60°C.
Fig. 15. ,, ,, ,, 30% at 10°C.
Fig. 16. ,, ,, ,, 20% at −10°C.
Fig. 17. ,, ,, ,, 20% at 50°C.
Fig. 18. ,, ,, ,, 10% at −10°C.
Fig. 19. ,, ,, ,, 10% at 0°C.
Fig. 20. ,, ,, ,, 5% at 0°C